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REFRACTORY ALLOY FOIL ROLLING DEVELOPMENT PROGRAM

Phase III Report
18 February 1963 - 20 May 1963

Metallurgical Processing Branch
Aeronautical Systems Division
Air Force Systems Command
United States Air Force
Wright-Patterson Air Force Base, Ohio

ASD Project No. 7-987

The processing of five alloys from ingot to 12" wide x 0.100" sheet is described. The five alloys are D-43 (Cb-10%W-1%Zr-0.1%C), B-66 (Cb-5%Mo-5%V-1%Zr), Cb-752 (Cb-10%W-2-1/2%Zr), Ta-10%W and T-111 (Ta-8%W-2%Hf). Small scale experiments on rolling of tungsten sheet are described.

(Prepared under Contract AF33(657)-8912 by E. I. Du Pont de Nemours & Company, Inc., Metals Center, Baltimore, Maryland)

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FOREWORD

This Interim Technical Documentary Progress Report covers the work performed under Contract AF33(657)-8912 from 18 February 1963 to 20 May 1963. It is published for technical information only and does not necessarily represent the recommendations, conclusions or approval of the Air Force.

This contract with E. I. Du Pont de Nemours & Company, Inc., Baltimore, Maryland was initiated under Manufacturing Methods Project 7-987, "Refractory Alloy Foil Rolling Development Program". It is being accomplished under the technical direction of Mr. H. L. Black of the Metallurgical Processing Branch, Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio.

Mr. John Symonds, Development Engineer, Metals Center, Baltimore, is the engineer directly responsible for the work. Others who cooperated in the work were Mr. J. S. Clark, Technical Supervisor and Mr. W. L. Patton, Process Development Engineer.

The primary objective of the Air Force Manufacturing Methods Program is to develop on a timely basis manufacturing processes, techniques and equipment for use in economical production of USAF materials and components. This program encompasses the following technical areas:

> Alloy Selection (Columbium, tantalum and tungsten alloys), Consolidation Techniques, Primary Breakdown, Rolling to Heavy Gauge Sheet, Foil Rolling.

Your comments are solicited on the potential utilization of the information contained herein as applied to your present or future production programs. Suggestions concerning additional Manufacturing Methods development required on this or other subjects will be appreciated.

PUBLICATION REVIEW

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ABSTRACT

The processing of three columbium-base alloys and two tantalum-base alloys from ingot to 0.100" thick x 12" wide sheet is described. The alloys were Cb-10%W-1%Zr-0.1%C (D-43), Cb-5%Mo-5%V-1%Zr (B-66), Cb-10%W-2-1/2%Zr (Cb-752), Ta-10%W, and Ta-8%W-2%Hf (T-111). The operations involved were: extrusion, hot-rolling, conditioning, annealing, and cold-rolling.

Rolling of small sheets of pure tungsten (0.060" thick and below) is described.

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I. INTRODUCTION

This report summarizes the work performed on Phase III of Contract No. AF33(657)-8912, entitled 'Refractory Alloy Foil Rolling Development Program'. The contract is sponsored by The Metallurgical Processing Branch ASRCT-1, AFSC Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio.

The program objective is to develop manufacturing processes for the production of tungsten, columbium and tantalum and/or their alloys in 24" wide foil, down to 0.001" thick. The program has been divided into five phases as follows:

1. The evaluation of the current state-of-the-art of refractory alloy foil rolling and the recommendation of the most promising alloys of tantalum, columbium, and tungsten for the remainder of the program.
2. The production and testing of ingots of columbium and tantalum alloys required for the manufacture of 12" wide foil and the investigation of the rolling of pure tungsten.
3. The production of coil blanks for each of the alloys selected and the evaluation of these coil blanks.
4. The rolling of foil in thicknesses from 0.001" - 0.005" and widths of 12" and the evaluation of the product.

5. The production of 24" wide coils of the approved alloys in the thickness range of 0.001" - 0.005".

At the conclusion of Phase I (State-of-the-Art Survey) the following compositions were selected for rolling to 12" wide foil:

1. Cb-10%W-1%Zr-0.1%C (D-43 alloy - previously designated X-110)
2. Cb-10%W-2-1/2%Zr (Cb-752 alloy)
3. Cb-5%Mo-5%V-1%Zr (B-66 alloy)
4. Ta-10%W
5. Ta-8%W-2%Hf (T-111 alloy)
6. Pure tungsten

Processing of the columbium and tantalum base alloys under this program commences with consolidation and proceeds through the various processing steps to foil as outlined under the five phases of the contract. It is expected that tungsten sheet will be purchased from a number of sources for rolling to foil.

The Phase III program described in this report consisted of processing of the 6" diameter columbium and tantalum base alloy ingots down to 0.1" thick, 12" wide sheet. The operations involved were: extrusion, conditioning, warm rolling to 5/8", heat treatment, and cold rolling to 0.1". Testing and evaluation procedures include: ultra-sonic testing of extruded sheet bars for soundness, chemical and spectrographic analyses, and metallography.

The tungsten portion of the program has been concerned with selection of starting material for rolling to foil, and small scale rolling trials using certain organic polymers as rolling lubricants.

II. SUMMARY

The objective of Phase III was to produce coil blanks (0.1" thick) in each of the columbium and tantalum base alloys for subsequent processing to foil. The objective in the tungsten portion of the program was to obtain the data necessary for predicting a process for producing 12" wide tungsten foil.

Extrusion - Columbium and Tantalum Alloys

The 6" diameter columbium base alloys (D-43, B-66, and Cb-752) were canned in mild steel and extruded to 4" x 1-1/2" sheet bar in the temperature range 2000°F-2100°F. The tantalum base alloys were extruded bare in the temperature range 3000°F-3100°F to 4" x 1-1/2" sheet bar. Soundness of the extrusions was checked ultrasonically.

The sheet bars were cut to 12" lengths and carefully surface conditioned.

Hot Rolling - Columbium and Tantalum Alloys

The Ta-10%W and T-111 alloy sheet bars were given a recrystallization heat treatment.

All the conditioned sheet bars were canned in mild steel for hot rolling. This was carried out at 2100°F on the 2-hi Schloemann Mill with 35" diameter rolls. The sheet bars were rolled perpendicular to the extrusion direction to approximately 5/8" thickness. The plates were de-canned, carefully conditioned and heat treated to give recrystallized structures.

Cold Rolling - Columbium and Tantalum Alloys

The annealed plates, approximately 1/2" thick, were cold rolled to 0.1" thickness on the Schloemann Mill 4-hi configuration (16" diameter work rolls). The 0.1" thick sheets had good surface quality. Gage uniformity was not considered adequate for continued rolling and has been corrected by a selective grinding operation.

The sheets have been annealed to give recrystallized microstructures.

Tungsten Processing

Small scale work on tungsten rolling from thicknesses up to 0.060" and at temperatures from room temperature to 300°F have indicated that tungsten sheet can be rolled to foil on D.M.C. equipment.

Certain organic polymer compounds have been found to have useful lubricating properties for rolling tungsten. These materials are stable up to 800°F and maintain lubricity at high unit loads.

III. EXTRUSION - COLUMBIUM & TANTALUM BASE ALLOYS

The three columbium alloy ingots were machined to 5.57" diameter and canned in 0.1" mild steel. A mild steel nose piece was integral with the can.

The two tantalum alloy ingots were machined to 5.7" diameter. A molybdenum nose piece was attached to each of these by drilling and tapping the ingot and nose piece and joining with a threaded molybdenum stud.

All ingots were extruded to 1-1/2" x 4" sheet bar (approximate 4.7:1 extrusion ratio) using glass lubrication. The original ingot top was the billet nose in each case.

Extrusion conditions are summarized in Table 1.

TABLE 1

EXTRUSION CONDITIONS FOR 6" DIAMETER COLUMBIUM BASE AND TANTALUM BASE ALLOY INGOTS

	Type Die	Extrusion Temp. °F	Pressure, ksi Break-thru	Pressure, ksi Running	Ram Speed in/sec.	Transfer time, sec.
D-43	Flat	2090	112	105	9	26
B-66	Flat	2080	134	126	10	27
Cb-752	Flat	1920	105	105	7	29
Ta-10%W	Cone ZrO_2 coated	3100	112	112	14	27
T-111	Cone ZrO_2 coated	3050	134	134	12	30

The steel cans on the three columbium alloy extrusions were stripped after extrusion. The can on the B-66 alloy ingot did not remain intact during extrusion and the sheet bar surface was marred by tears, typically 1/8" deep. The front end of the Cb-752 alloy extrusion was split to a length of 4".

The extruded lengths were ultrasonically checked and sawed into 12" lengths (for cross rolling to 12" wide foil) and smaller pieces for test purposes.

The as-extruded microstructures are shown in Figures 1, 2, 3, 4, and 6. Hardness measurements were made on as-extruded transverse sections. The ranges of hardnesses for the five extrusions are shown in Table 2.

FIGURE 1

(a)

D-43 alloy

As-extruded micro-structure

Longitudinal-front of extrusion

X100

(b)

D-43 alloy

As-extruded micro-structure

Longitudinal-back of extrusion

X100

FIGURE 2

(a)

B-66 alloy

As-extruded microstructure

Longitudinal-front of extrusion

X100

(b)

B-66 alloy

As-extruded microstructure

Longitudinal-back of extrusion

X100

FIGURE 3

(a)

Cb-752 alloy

As-extruded micro-
structure

Longitudinal-front
of extrusion

X100

(b)

Cb-752 alloy

As-extruded micro-
structure

Longitudinal-back
of extrusion

X100

FIGURE 4

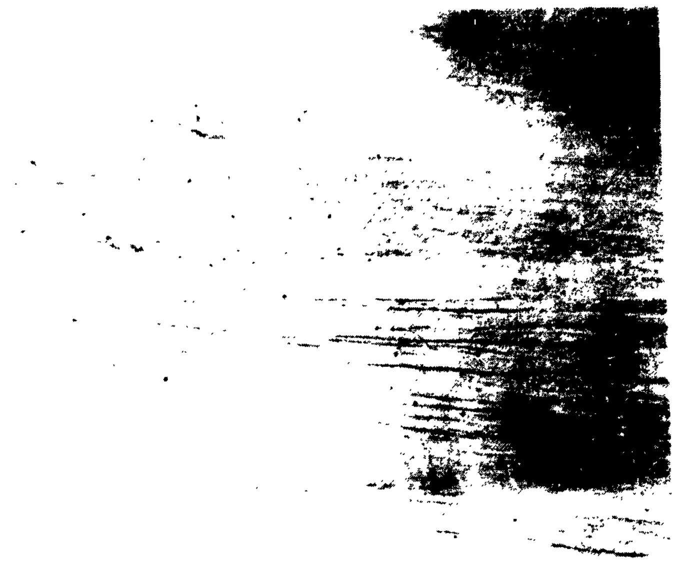
(a)

Ta-10%W alloy

As-extruded microstructure

Longitudinal-front of extrusion

X100



(b)

Ta-10%W alloy

As-extruded microstructure

Longitudinal-back of extrusion

X100

FIGURE 5

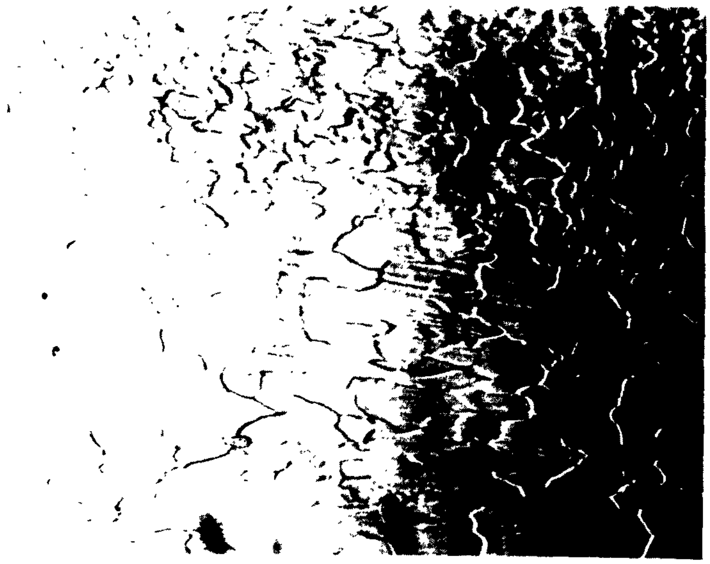
(a)

Ta-10%W alloy

Microstructure after extrusion and 1 hour at 2700°F.

Longitudinal-front of extrusion

X100



(b)

Ta-10%W alloy

Microstructure after extrusion and 1 hour at 2700°F.

Longitudinal-back of extrusion

X100

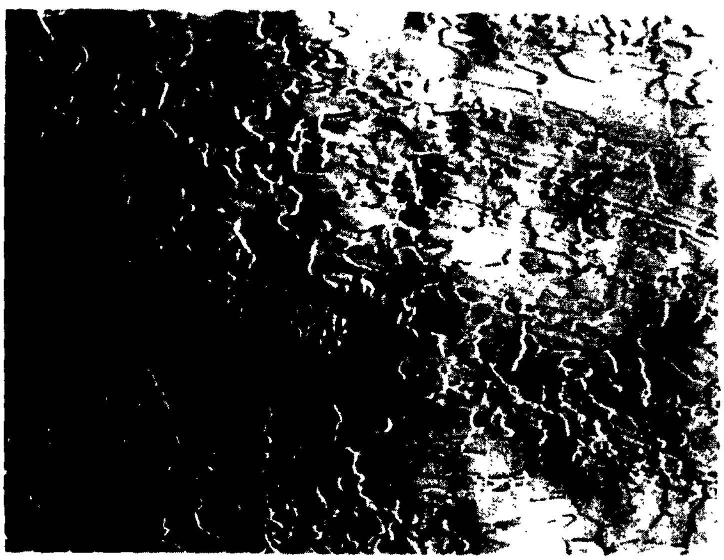


FIGURE 6

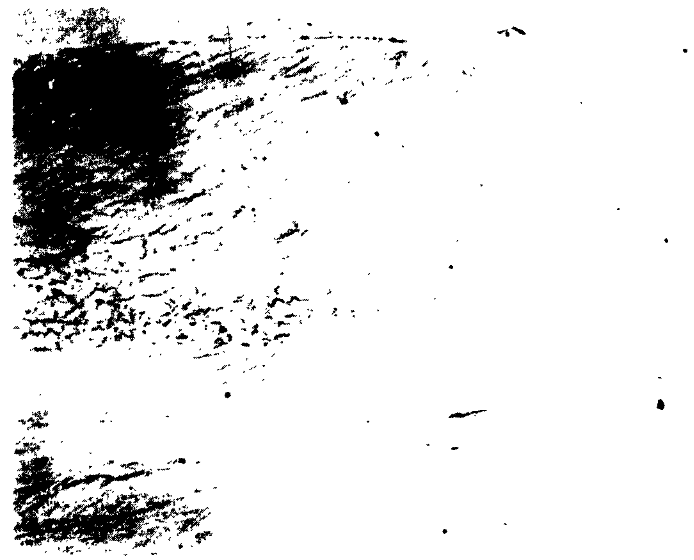
(a)

T-111 alloy

As-extruded micro-
structure

Longitudinal-front
of extrusion

X100



(b)

T-111 alloy

As-extruded micro-
structure

Longitudinal-back
of extrusion

X100

FIGURE 7

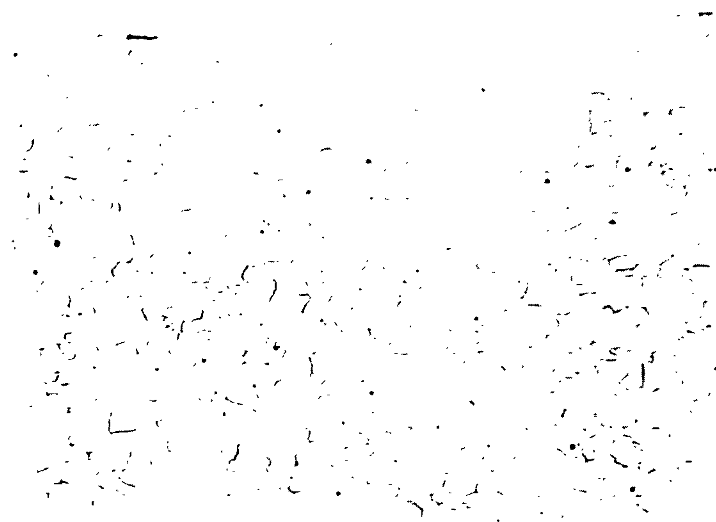
(a)

T-111 alloy

Microstructure after extrusion and 1 hour at 2700°F.

Longitudinal-front of extrusion

X100



(b)

T-111 alloy

Microstructure after extrusion and 1 hour at 2700°F.

Longitudinal-back of extrusion

X100

TABLE 2

HARDNESS READINGS OF AS-EXTRUDED SHEET BARS*

	R_A	R_B
D-43 alloy, Front	57.2 - 60.2	89.9 - 93.5
Back	58.4 - 60.2	88.9 - 95.0
B-66 alloy, Front	61.0 - 63.2	94.0 - 96.0
Back	60.2 - 62.0	96.0 - 98.0
Cb-752 alloy, Front	59.8 - 62.0	89.5 - 93.2
Back	59.8 - 62.0	91.1 - 93.9
Ta-10%W alloy, Front	62.5 - 64.0	99.5 - 100.7
Back	65.7 - 66.3	101.7 - 102.5
T-111 alloy, Front	62.0 - 64.0	98.1 - 99.8
Back	62.6 - 64.3	97.0 - 103.0

*Independent readings on Rockwell A and B scales.

Since most of the sheet bars were slightly bent they were flattened on a 2000 ton forge press (after pre-heating to 2000°F). Flat sheet bars were required because conditioning of the major surfaces of all sheet bars was accomplished by machining rather than spot grinding. (An unusually extensive conditioning schedule is being used on these sheet bars in order to ensure good surface condition in the later stages of processing). The sides of sheet bars were conditioned by hand grinding.

Chemical analyses were made on the back and front of each extrusion. The results of these analyses are summarized in Table 3.

TABLE 3

ANALYTICAL DATA ON Cb- AND Ta-BASE ALLOY EXTRUDED SHEET BARS

Alloy	%W	%Zr	%V	%Mo	%Hf	Interstitials, ppm O	N	C	H
D-43, Front	9.1	0.90	-	-	-	56	36	1000	6
Back	9.3	0.91	-	-	-	78	38	950	2
Specification (1)	9-11	0.75-1.25	-	-	-	400 max.	100 max.	800-1200	20 max.
B-66, Front	-	0.83	5.1	4.8	-	127	18	72	7
Back	-	0.88	5.4	5.0	-	132	38	74	9
Specification (2)	-	0.85-1.3	4.5-5.5	4.5-5.5	-	300 max.	200 max.	200 max.	-
Cb-752, Front	9.4	2.2	-	-	-	127	19	63	10
Back	9.5	2.3	-	-	-	136	15	64	5
Specification (3)	9-11	2-3	-	-	-	400 max.	100 max.	100 max.	20 max.
Ta-10%W, Front	9.3	-	-	-	-	100	31	40	10
Back	10.0	-	-	-	-	138	37	24	17
Specification (4)	9-11	-	-	-	-	100 max.	50 max.	50 max.	10 max.
T-111, Front	8.4	-	-	-	1.81	77	39	35	10
Back	8.3	-	-	-	1.85	94	35	34	19

(1) Tentative Du Pont Specification

(2) Westinghouse Special Technical Data Sheet 52-364

(3) North American Aviation, Inc. Material Specification LBO170-176

(4) Tentative National Research Corporation Specification

Semi-quantitative spectrographic analyses of the alloying elements were obtained at four points across the half-widths of each sheet bar as shown in Figure 8. The results are tabulated in Table 4.

TABLE 4

SEMI-QUANTITATIVE SPECTROGRAPHIC ANALYSES ON Cb- AND Ta-BASE ALLOY EXTRUDED SHEET BARS

Alloy	Alloy Element	Alloy Content % Edge 1	2	3	Center 4
D-43, Front	W	10.4	9.5	9.8	10.0
	Zr	0.9	1.0	1.0	1.0
Back	W	10.3	9.6	10.0	9.9
	Zr	1.0	1.0	1.0	1.0
B-66, Front	Mo	5.0	5.0	4.9	5.0
	V	5.0	5.0	5.0	5.0
	Zr	1.0	1.0	1.0	1.0
Back	Mo	5.0	5.0	5.0	4.9
	V	4.5	5.3	5.0	5.1
	Zr	0.9	1.0	1.0	1.0
Cb-752, Front	W	10.7	10.1	9.7	9.5
	Zr	2.5	2.5	2.5	2.5
Back	W	9.6	9.9	9.6	11.2
	Zr	2.6	2.5	2.4	2.5
Ta-10%W, Front	W	10.6	9.4	10.6	10.6
Back	W	12.0	10.5	9.0	8.0
T-111, Front	W	8.5	8.1	7.9	7.6
	Hf	2.0	1.9	2.0	2.1
Back	W	8.0	7.7	7.9	7.1
	Hf	2.0	2.0	1.9	2.1

Since spectrographic analyses have not been standardized for absolute values of the alloying elements in these alloys, Table 4 can only be used to illustrate the variation in alloy element analyses from point to point. These analyses show the tungsten content in the back of the Ta-10%W alloy extrusion varying from 8% to 12%. This variation is difficult to explain in view of the previous homogeneity (spectrographic and microprobe) checks on the ingot. Subsequent analyses on Ta-10%W Sheet #3 will yield more information on the degree of variation.

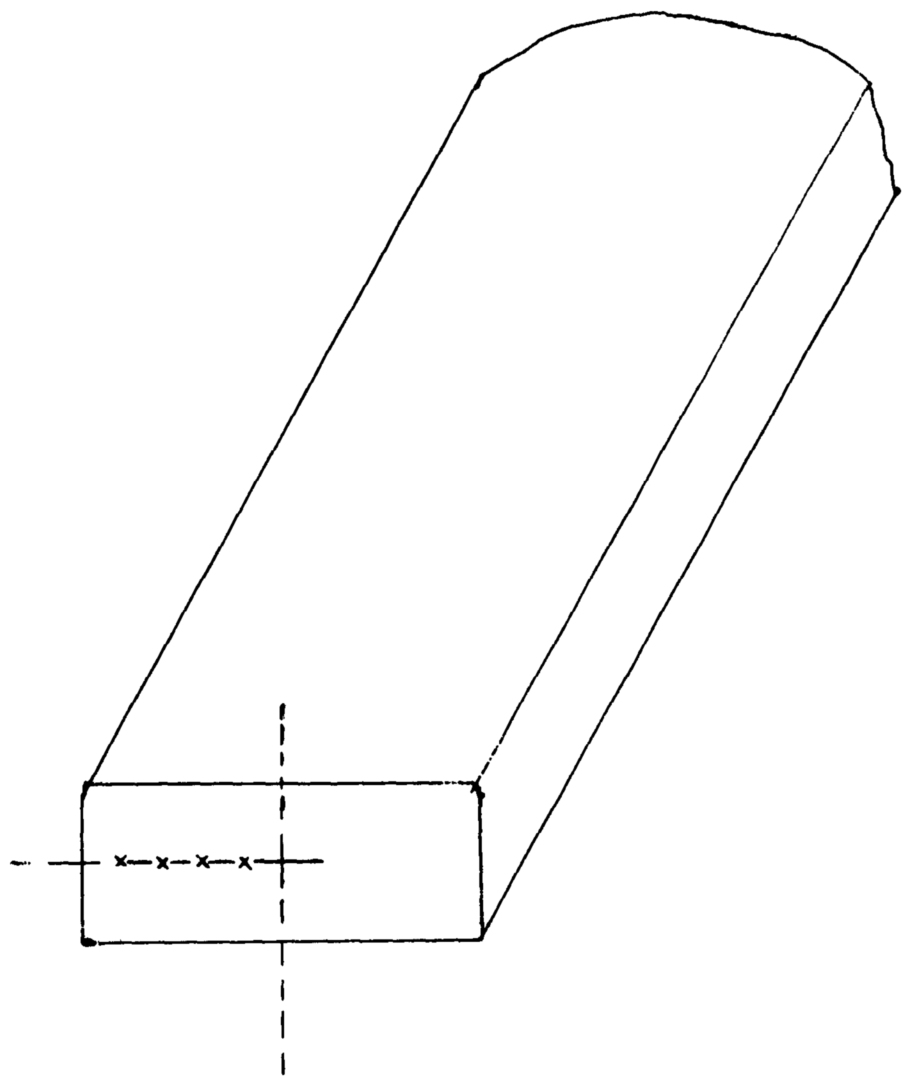


FIGURE 8

LOCATIONS OF SPECTROGRAPHIC ANALYSES ON EXTRUDED SHEET BARS

IV. HOT ROLLING - COLUMBIUM & TANTALUM BASE ALLOYS

The Ta-10%W and T-111 alloy sheet bars were recrystallized by a one hour 2700°F vacuum heat treatment. The recrystallized microstructures are shown in Figures 5 and 7. The hardness of the Ta-10%W alloy decreased from 99-103 R_B (as-extruded) to 93-96 R_B; that of the T-111 alloy decreased from 97-103 R_B (as-extruded) to 92-94 R_B.

The sheet bars varied in thickness from 1-3/32" (for the B-66 alloy) to 1-15/32" (for the T-111 alloy) after conditioning. They were canned in 1/8" thick mild steel for rolling.

The sheet bars were rolled to approximately 1/2" thick plate on the Schloemann Mill at D.M.C., Baltimore, using 2-hi (35" diameter) rolls. Furnace pre-heat temperature was 2100°F. Rolling direction was at right angles to the extrusion direction. The percentage reductions effected on each of the sheet bars were:

		Reduction
D-43	Sheet bar - 1	58%
	2	53%
	3	52%
B-66	Sheet bar - 1	44%
	2	46%
	3	54%
Cb-752	Sheet bar - 1	55%
	2	58%
Ta-10%W	Sheet bar - 1	58%
	2	62%
	3	61%
T-111	Sheet bar - 1	59%
	2	57%

After rolling, the mild steel cans were removed by pickling. The Cb alloy plate surfaces were uneven and contained a few areas of fine crazing but did not have any deep cracks or defects. The Ta alloy plate surfaces were crazed with fissures approximately 30 mils deep.

Heavy conditioning on all plate surfaces was then carried out by grinding. The extent of this conditioning can be seen in Figures 9 - 13 which show one conditioned plate in each of the alloys.

The processing route used from sheet bar to 0.100" sheet was found to be satisfactory (with respect to conservation of material, surface quality, and shape of final sheet). However, this route will not be applicable to all refractory alloys; after conditioning the 1/2" thick plates by grinding out localized defects, the plates varied in thickness by as much as 0.045". This put large demands on the ductility of the plates in subsequent cold rolling because of the variation in reduction from point to point in the first one or two passes.

Much work remains to be done in optimizing the breakdown route for sheet production. Some of the possibilities requiring exploration are:

1) In-process coatings for hot rolling to minimize contamination and conditioning losses.
2) Certain refractory alloys may have sufficient ductility to be rolled cold or in the range 500°F - 1000°F from the sheet bar stage. Rolling at low temperatures could result in

smoother as rolled surfaces, thus minimizing conditioning losses. This presupposes the availability of a mill capable of heavy reductions in the thick material.

3) Also, depending on ductility, roller leveling of heavy plate may be practicable. This would make it possible to condition by belt grinding or even machining, thus obtaining the optimum in shape and surface condition.

FIGURE 9

HOT ROLLED AND CONDITIONED D-43 ALLOY PLATE

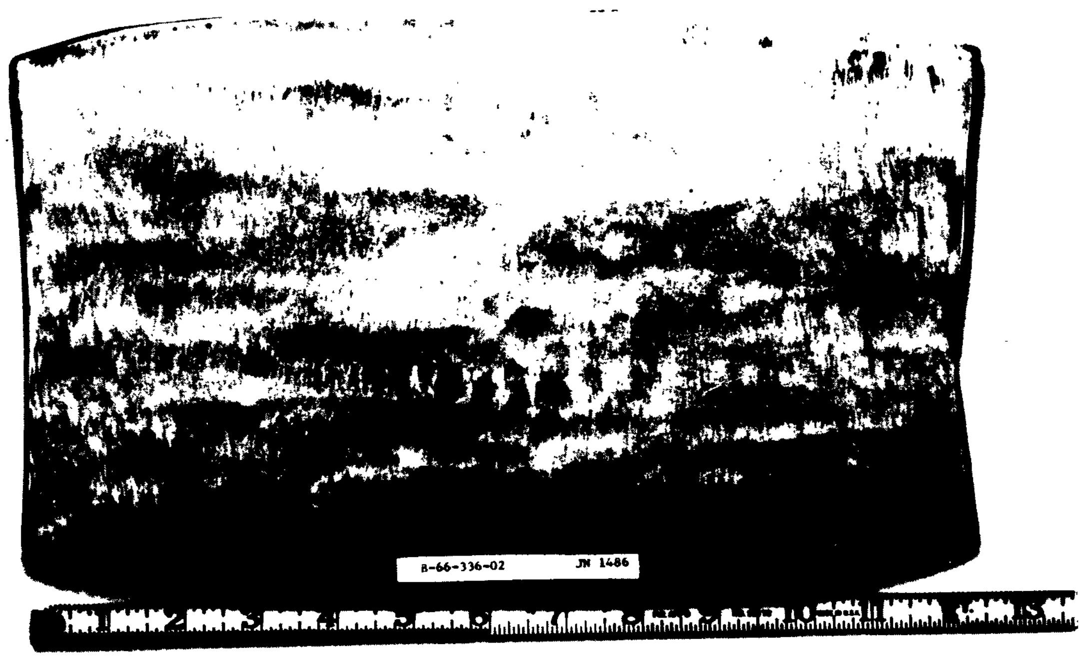


FIGURE 10

HOT ROLLED AND CONDITIONED B-66 ALLOY PLATE

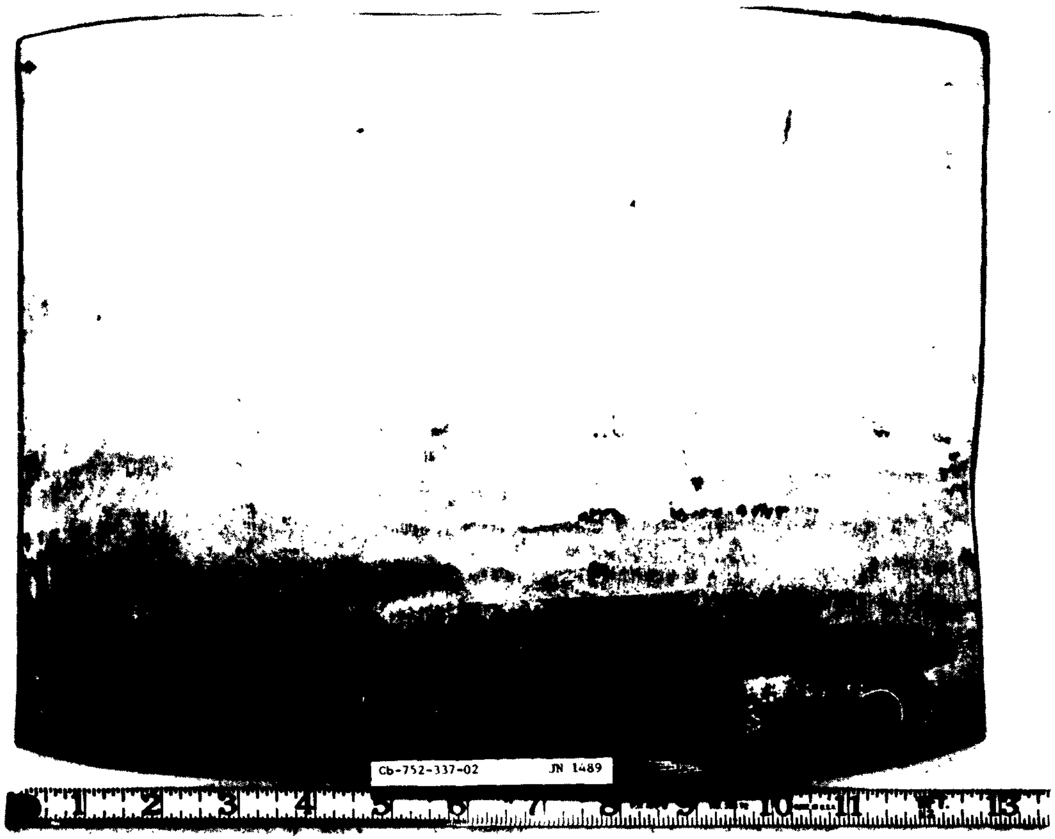
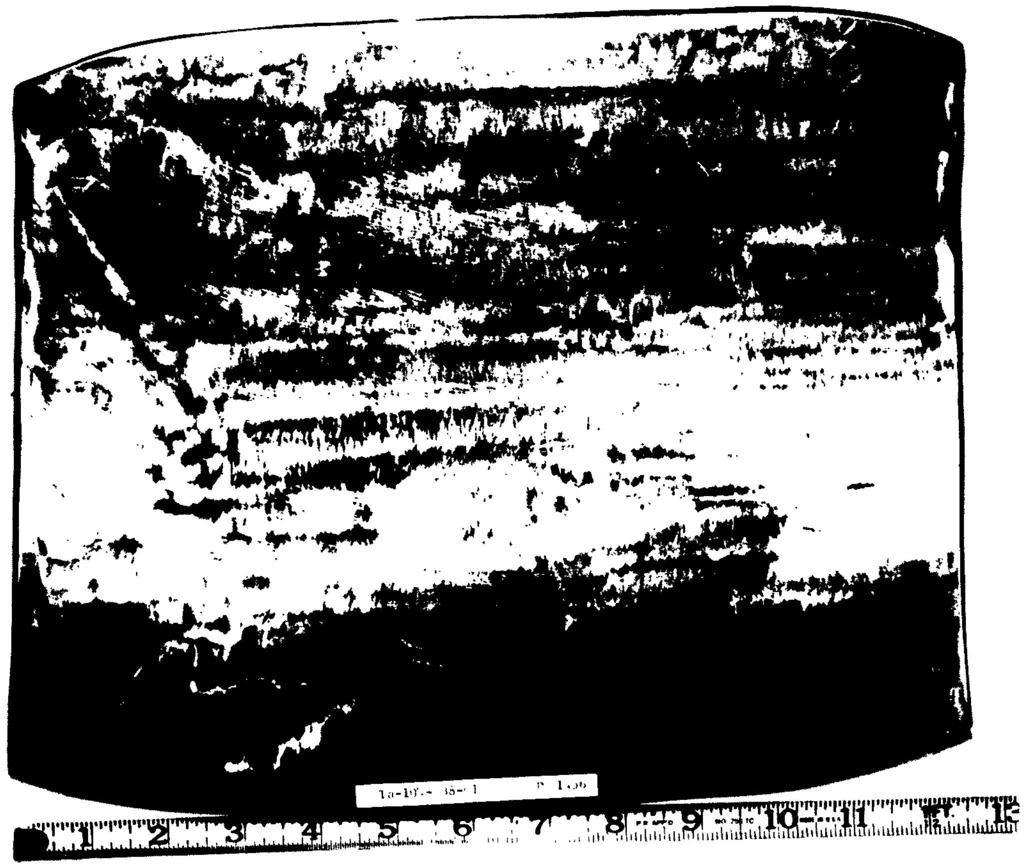


FIGURE 11

HOT ROLLED AND CONDITIONED Cb-752 ALLOY PLATE



<u>FIGURE 12</u>

HOT ROLLED AND CONDITIONED Ta-10%W ALLOY PLATE

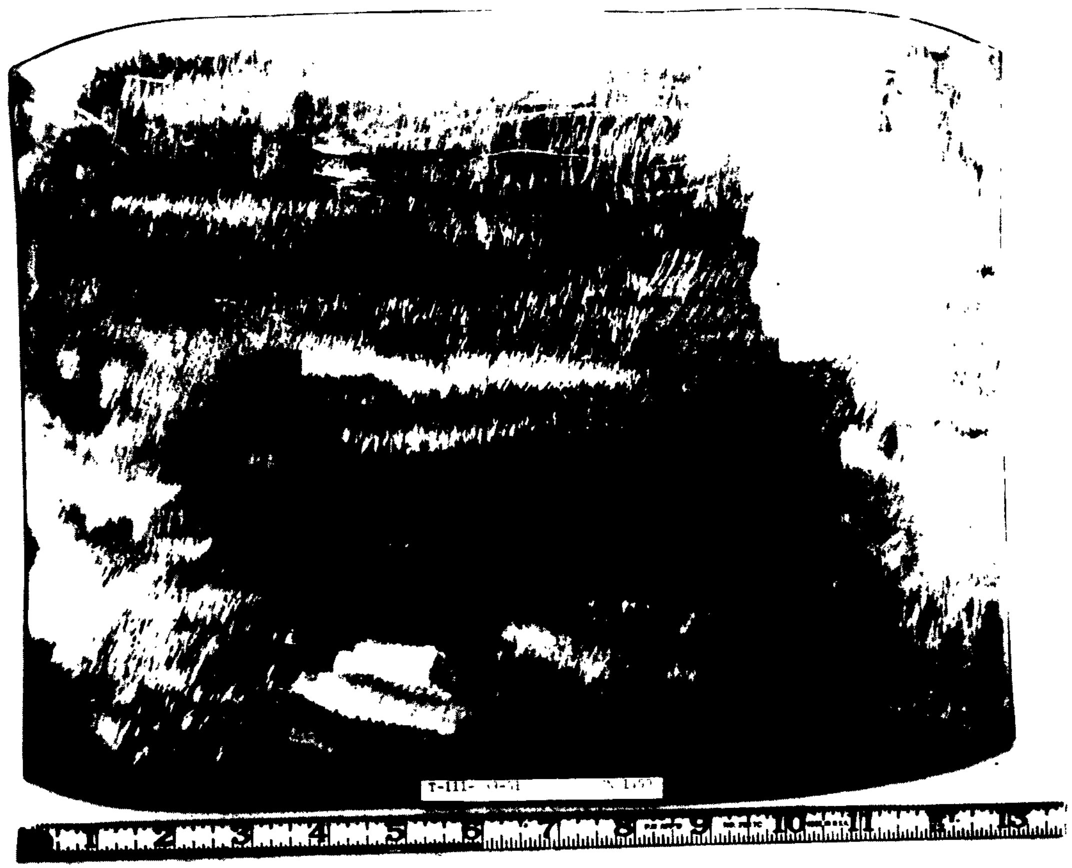


FIGURE 13

HOT ROLLED AND CONDITIONED T-111 ALLOY PLATE

Plate Heat Treatment

The conditioned 1/2" thick plates were heat treated as indicated in Table 5, which also shows hardnesses before and after heat treatment.

TABLE 5

HEAT TREATMENT OF COLUMBIUM AND TANTALUM ALLOY PLATES

Plate Identity	Heat Treatment	Hardness (R_A)	
		Before	After
D-43-1	2500°F, 1 hour	58-61	50
D-43-2, 3	2600°F, 1 hour	-	-
B-66-1, 2, 3	2500°F, 1 hour	63-65	56
Cb-752-1	2600°F, 1 hour	60-61	50
Cb-752-2	2500°F, 1 hour	-	-
Ta-10%W-1, 2, 3	2600°F, 1 hour	63-65	56
T-111-1, 2	2600°F, 1 hour	65-67	54

Typical heat treated microstructures are shown in Figures 14 - 18.

Figure 14

D-43 alloy plate (No. 1) micro-structure

Hot-rolled + 1 hour at 2500°F.

Longitudinal

X100

Figure 15

B-66 alloy plate (No. 1) micro-structure

Hot-rolled + 1 hour at 2500°F.

Longitudinal

X100

Figure 16

Cb-752 alloy plate (No. 1) microstructure

Hot-rolled + 1 hour at 2600°F.

Longitudinal

X100

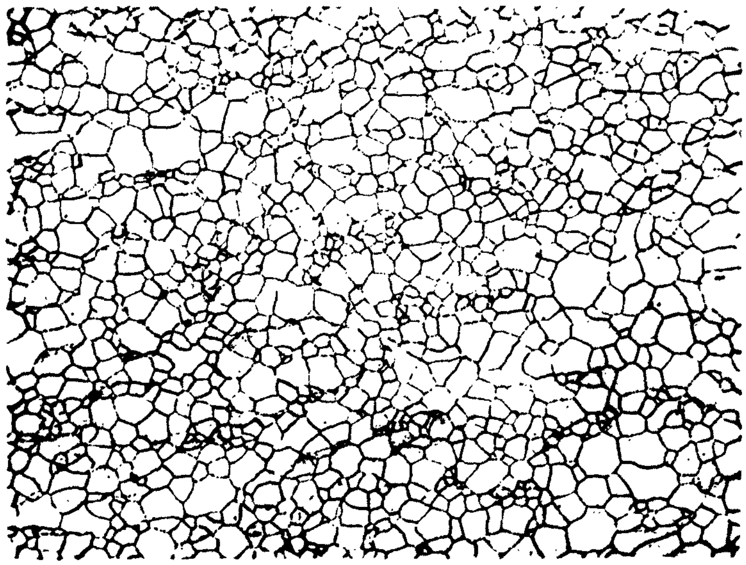


Figure 17

Ta-10%W alloy plate (No. 2) microstructure

Hot-rolled + 1 hour at 2600°F.

Longitudinal

X100

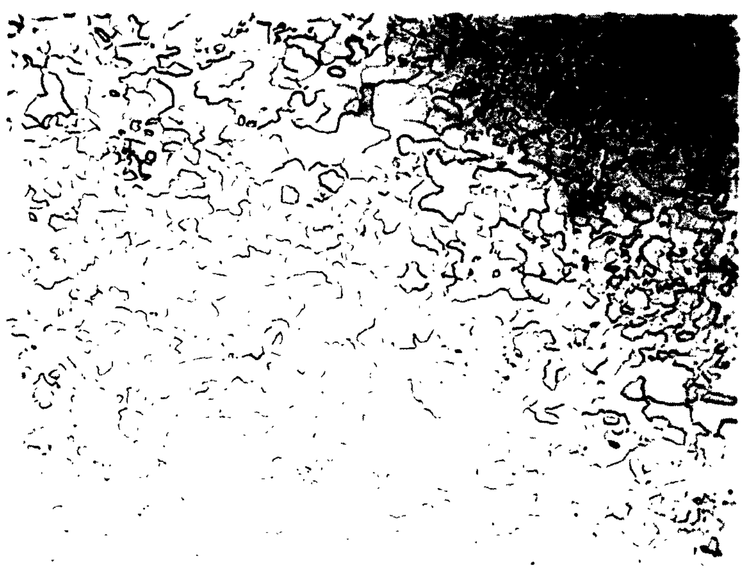


FIGURE 18

T-111 alloy plate (No. 2) microstructure

Hot-rolled + 1 hour at 2600°F. Longitudinal

X100

V. COLD ROLLING - COLUMBIUM AND TANTALUM BASE ALLOYS

The approximate dimensions and the weights of each of the conditioned and heat treated plates are shown in Table 6.

TABLE 6

DIMENSIONS AND WEIGHTS OF COLUMBIUM AND TANTALUM ALLOY PLATES

	Dimensions (Length x Width x Thickness)	Weight Lbs.
D-43-1	10-3/4" x 12-5/16" x (0.394"-0.397")	17.0
2	8-3/4" x 12-5/8" x (0.490"-0.507")	17.5
3	8-1/2" x 12-3/8" x (0.495"-0.507")	17.0
B-66-1	7-3/8" x 12-1/2" x (0.469"-0.488")	13.6
2	7-1/2" x 12-1/4" x (0.482"-0.505")	13.5
3	7-5/8" x 12-1/4" x (0.508"-0.516")	14.3
Cb-752-1	9-1/4" x 12-1/2" x (0.442"-0.457")	17.0
2	10-1/8" x 12-1/2" x (0.423"-0.430")	16.9
Ta-10%W-1	10-1/4" x 12-1/4" x (0.437"-0.460")	33.4
2	10-1/4" x 12-1/8" x (0.398"-0.440")	31.3
3	10" x 12-1/4" x (0.493"-0.498")	36.4
T-111-1	9-3/4" x 12-1/2" x (0.459"-0.467")	34.6
2	10-1/4" x 12-11/16"x (0.488"-0.499")	38.8

The plates were cold rolled to 0.100" on the D.M.C. Schloemann Mill 4-hi set-up (16" diameter work rolls). Rolling direction was the same as for the hot rolling, i.e., perpendicular to the extrusion direction. Rolling was interrupted at approximately 1/4" thickness to examine the plates and spot condition any surface defects which had developed in rolling.

Part of one B-66 plate (B-66-2) was lost during rolling; on the initial pass a crack propagated from the thinner side (this thickness variation was caused by the uneven removal of metal in conditioning, corresponding to uneven distribution of surface defects after hot rolling) to a distance of one-third the plate width. The cracked portion of the plate was sawn off and rolling resumed. In order to obtain the necessary 12" width it was necessary to cross roll; this was done in alternate passes until the desired width was attained after which all rolling was in the direction perpendicular to the original extrusion direction. No further cracking occurred in rolling by this revised schedule.

Sheets of excellent surface quality were obtained; the only surface defects occurred on one side of one sheet of T-111 alloy on which a few areas of very fine surface checking (approximately 1 mil deep) were observed, probably caused by incomplete conditioning at the plate stage. Light belt conditioning removed this defect. The approximate dimensions and weights of the cold rolled, pickled, and side-trimmed sheets are given in Table 7.

TABLE 7

DIMENSIONS OF COLD ROLLED COLUMBIUM AND TANTALUM ALLOY SHEETS*

D-43-1	44" x 12-1/2" x 0.0925"
2	43-1/2" x 12-1/2" x 0.0960"
3	41-1/2" x 12-1/2" x 0.1020"
B-66-1	36" x 12-1/2" x 0.0965"
2	26-1/4" x 11-1/2" x 0.0960"
3	42" x 12" x 0.0965"
Cb-752-1	44" x 13" x 0.0910"
2	43" x 12-1/2" x 0.0900"
Ta-10%W-1	46" x 12" x 0.0980"
2	42-3/4" x 12" x 0.1000"
3	46-1/2" x 12-1/2" x 0.1035"
T-111-1	49-1/2" x 13" x 0.1025"
2	44" x 12-1/2" x 0.1020"

*Length dimensions include 'fish-tails'. Before surface grinding.

Gage uniformity of the sheets was not considered to be adequate for further rolling, bearing in mind the unusually stringent requirements of foil rolling. Edge-to-center measurements showed variation in the range 2 - 6 mils. (Most of this variation occurred in the 1/2" - 3/4" wide bands along the edges of the sheet). Longitudinal measurements on individual sheets varied 0 - 6 mils. The sheets have been roller-leveled and belt-ground (to improve gage uniformity) before resumption of rolling. Figure 19 shows five sheets after roller leveling and grinding. These five sheets correspond to the conditioned 1/2" plates shown in Figures 9 - 13. The detailed inspection reports on gage variation and shape are shown in Appendix II.

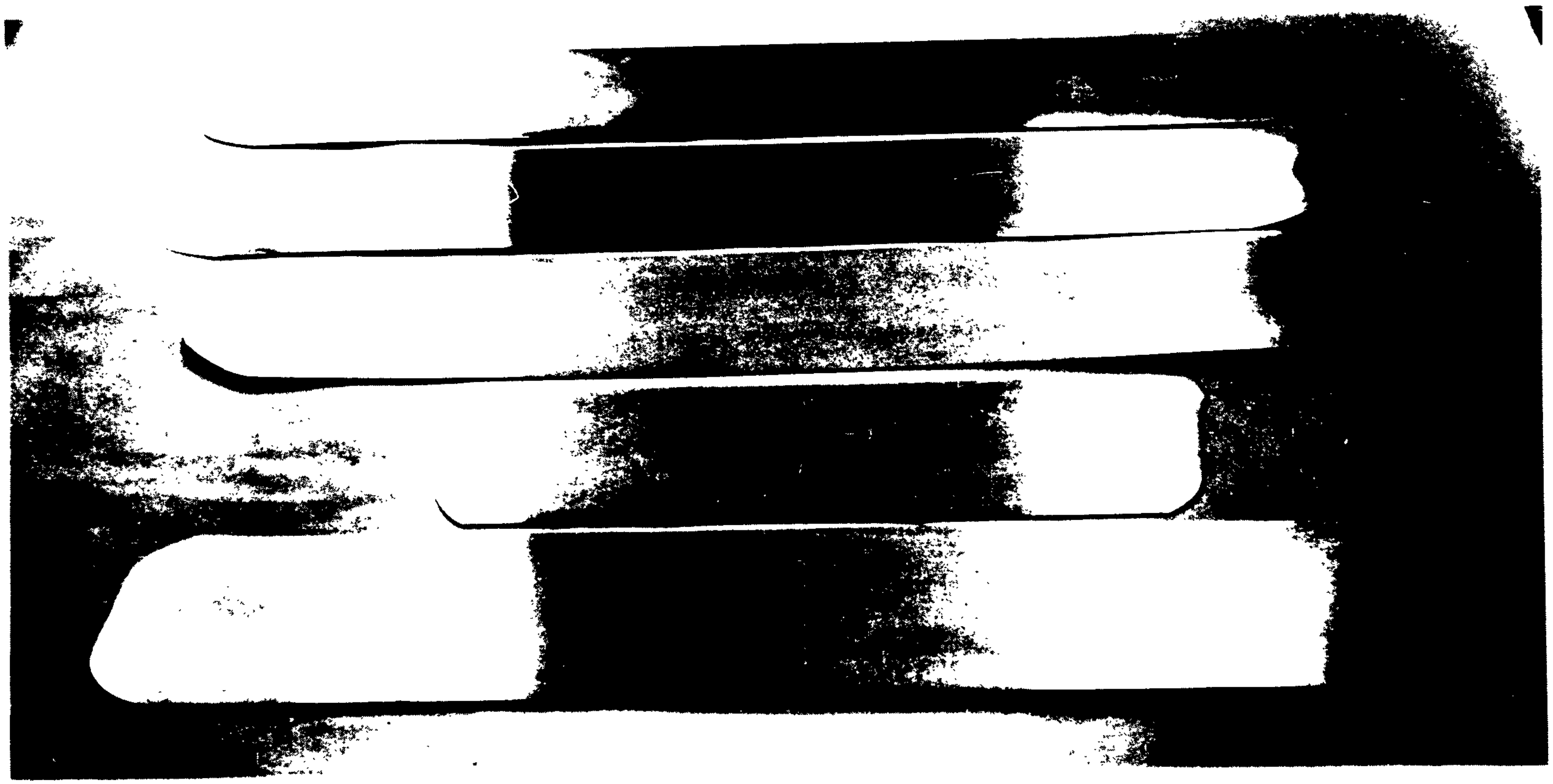


FIGURE 19

0.100" sheets after

roller-leveling and belt-sanding

T-111-1
Ta-10%W-2
Cb-752-2
B-66-2
D-43-2

The sheets were heat treated as indicated in Table 8, which also includes heat treated hardness values.

TABLE 8

HEAT TREATMENT OF COLD-ROLLED COLUMBIUM & TANTALUM ALLOY SHEETS

Sheet Identity	Heat Treatment	Heat Treated Hardness (R_A)
D-43-1	2400°F, 1 hour	46-49
D-43-2, 3	2500°F, 1 hour	49-50
B-66-1, 2, 3	2400°F, 1 hour	58
Cb-752-1	2500°F, 1 hour	53-54
Cb-752-2	2400°F, 1 hour	-
Ta-10%W-1, 2, 3	2500°F, 1 hour	59-60
T-111-1, 2	2500°F, 1 hour	59-60

Microstructures of the annealed sheets are shown in Figures 20 - 26.

Transverse room temperature tensile tests were carried out on one sheet of each alloy, the results of which are reported in Table 9.

Figure 20

0.100" sheet

D43-1

Cold-rolled, 1 hour
at 2400°F.
Grain size: ASTM 7-8

X100

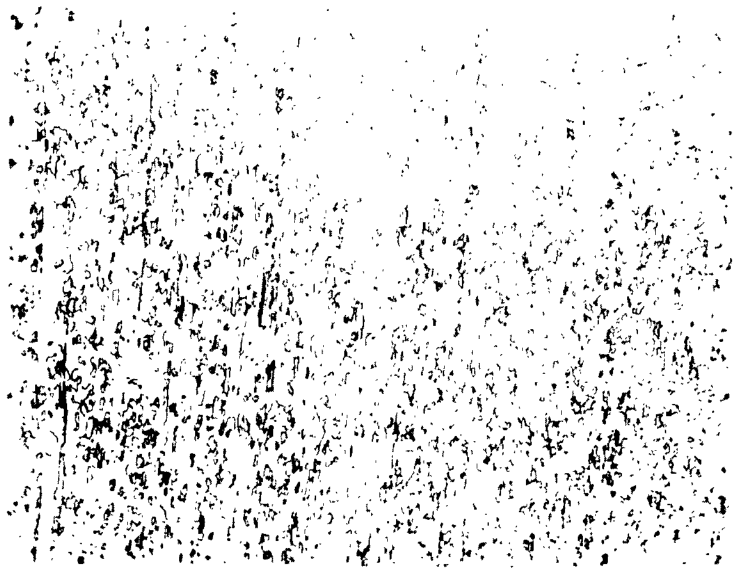


Figure 21

0.100" sheet

D43-2

Cold-rolled, 1 hour
at 2500°F.
Grain size: ASTM 7-8

X100

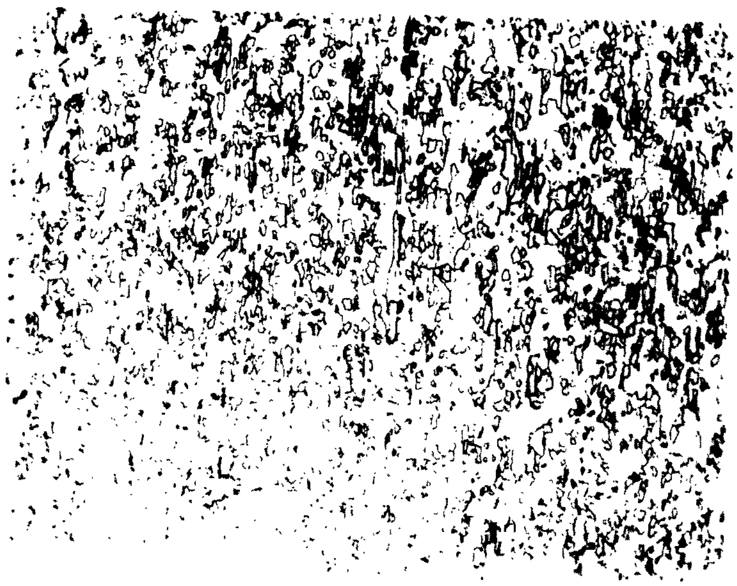


Figure 22

0.100" sheet

B66-1

Cold-rolled, 1 hour
at 2400°F.
Grain size: ASTM 5-6

X100

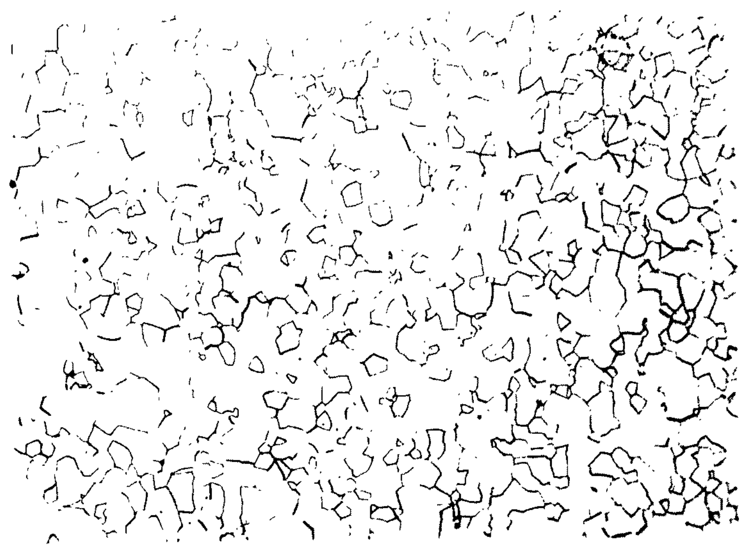


Figure 23

0.100" sheet

Cb-752-1

Cold-rolled, 1 hour
at 2500°F.
Grain size: ASTM 6-8

X100

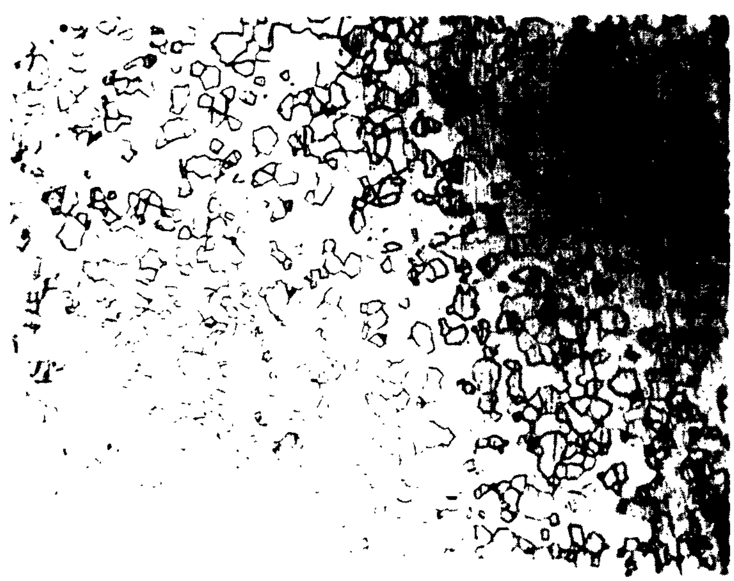


Figure 24

0.100" sheet

Cb-752-2

Cold-rolled, 1 hour
at 2400°F.
Grain size: ASTM 6-8

X100

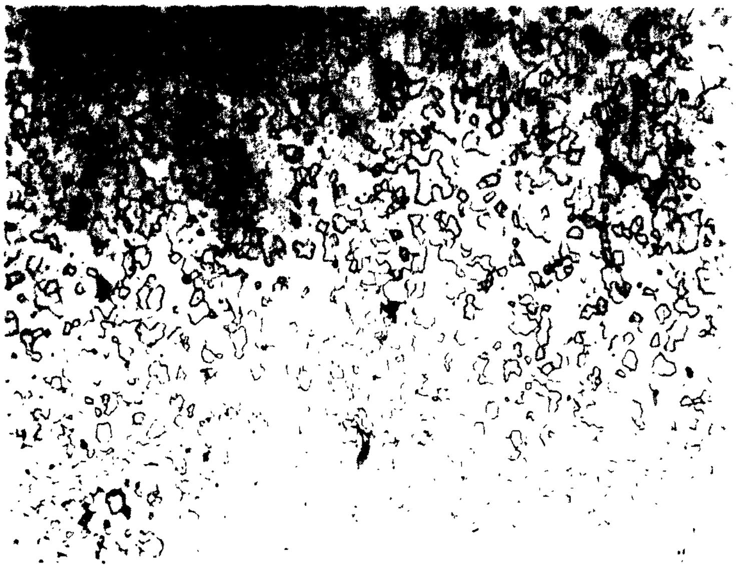


Figure 25

0.100" sheet

Ta-10%W-2

Cold-rolled, 1 hour
at 2500 F.
Grain size: ASTM 8

X100

Figure 26

0.100" sheet

T111-2

Cold-rolled, 1 hour at 2500°F.
Grain size: ASTM 7-8

X100

TABLE 9

TRANSVERSE ROOM TEMPERATURE TENSILE TESTS ON 0.100" ANNEALED SHEETS

Alloy	Sheet Identity	U.T.S. x 1000 psi	Y.S. (0.2%) x 1000 psi	Elong. % in 1"
D-43	3	71.1	44.9	35
B-66	1	95.5	72.8	34
Cb-752	1	88.7	65.7	31
Ta-10%W	2	98.4	95.7	34
T-111	2	102.9*	102.9	34

Specimens were pulled at 0.005"/in./min. to yield and then 0.05"/in./min. to ultimate.

*This specimen elongated faster than 0.05"/in./min. could apply load.

VI. TUNGSTEN

It was decided in Phase I of this program and concurred in by the Air Force, that tungsten sheets should be purchased for rolling to foil and that Du Pont should work only with techniques for rolling the relatively thin gage tungsten.

The small scale work carried out to-date has been concerned with selection of the best starting materials for rolling to foil and with tests on special rolling lubricants which appear to be particularly suitable for rolling of thin gage tungsten.

The test work undertaken so far has been carried out on a Fenn 4-hi mill with 3-1/4" diameter work rolls. Small sheets of tungsten up to 6" in width and 0.060" and below in thickness have been rolled on this mill in order to evaluate tungsten sheet (with respect to rollability) from various sources. The technique used has been to roll the sheet in the temperature range room temp. - 350°F. For this study the work rolls were heated by a gas burner installation on the mill.

Prior work by Du Pont had indicated that certain organic polymers have attractive properties for use as rolling lubricants. Two classes of polymer are of particular interest; these (identified by their Du Pont commercial names) are:

1. A polytetrafluoryl-ethylene compound developed by the Du Pont Organic Chemicals Dept. and known as 'Vydax'. It is used as a dispersion in Freon and can be sprayed on the metal to be rolled.

2. A poly-imide compound developed by the Du Pont Film Dept. and applied as a clear transparent film. The Du Pont designation is 'H'-film.

These two lubricants are stable at temperatures up to 800°F and they maintain lubricity under heavy unit loadings. The 'Vydax' type lubricant is more suitable for the immediate work on tungsten. While the 'H'-film shows excellent lubricating properties (it also acts as a thermal insulator in rolling and prevents chilling of the metal by the work rolls) a problem remains in that the film wrinkles during rolling and imparts dents to the sheet being rolled.

The technique involved in the use of 'Vydax' lubrication is uncomplicated. The lubricant is applied to the sheet surface by spraying, dipping, or brushing. The Freon vehicle evaporates rapidly leaving a waxy film. The sheet may be preheated after lubricant application. Removal of the lubricant prior to annealing is effected by a 3-4 minute pickle in a weak HNO_3-HF solution with a surfactant addition.

Materials

The materials used in the small scale work may be divided into three categories:

1. Commercial powder metallurgy sheet from two sources. Processing histories of these sheets were not available, but both materials were believed to have been compacted from lightly 'doped' powder.

2. Powder metallurgy sheet (0.060") made from undoped powder by Fansteel Metallurgical Corporation under their Navy Tungsten Sheet Program (Contract NOw-60-0621-c).
3. Sheet made from arc-cast tungsten by Universal Cyclops Steel Corporation and produced in a similar manner to that described by this company in the reports under the Air Force Tungsten Sheet Rolling Program (Contract AF33(600)-41917).

We are unable to make a comparison of the above materials with respect to 'rollability'. In the course of this work, technique has been gradually improved and is thus, not a constant. Some of these materials have been successfully rolled from 0.060" at a temperature of approximately 300°F. At 0.020" thickness and below, certain of the materials have been rolled up to 55% reduction at room temperature and up to 80% reduction at 250°F (without intermediate stress relief). Edge trimming is usually necessary to remove edge cracks at some intermediate point when making heavy reductions. Further screening work is necessary to select the best starting material for this contract. Some tentative data on the small scale tungsten sheet rolling experiments are summarized in Table 10. The extremely heavy reductions made on Universal Cyclops arc-cast sheet with moderate separating force are noteworthy. No explanation for the comparative ease of rolling this material is apparent.

It is considered feasible to roll 12" wide tungsten foil 0.005" thick and below on the Schloemann Mill. Twelve inch wide tungsten sheet will be purchased in Phase IV of this contract and rolled at D.M.C.

TABLE 10

TENTATIVE DATA ON ROLLING TUNGSTEN SHEET

Type Sheet	Initial Thickness	Pass No.	% Reduction (Cumulative)	Temperature °F Rolls	Temperature °F Pre-heat(2)	Lubricant	Mill separating force/in. width x 1000 lb.
1(1)	0.019	1	21	R.T.	R.T.	None	125
		2	32	R.T.	R.T.	None	132
		3	37	R.T.	R.T.	None	140
		4	41	R.T.	R.T.	None	152
1	0.019	1	36	R.T.	R.T.	PR-201(3)	132
		2	54	R.T.	R.T.	PR-201	162
		3	58	R.T.	R.T.	PR-201	182
1	0.019	1	34	250	500	None	134
1	0.019	1	46	250	500	PR-201	130
1	0.019	1	34	350	800	PR-201	37
		2	55	350	800	PR-201	50
1	0.019	1	21	R.T.	R.T.	Mineral Oil(4)	90
		2	24	R.T.	R.T.	Mineral Oil	95
		3	31	R.T.	R.T.	Mineral Oil	95
2(1)	0.061	1	33	275	800	'H'-Film	40
		2	57	275	800	'H'-Film	50
3(1)	0.025	1	83	300	500	PR-201	65
3	0.023	1	80	300	500	PR-201	55

(1) Numbers refer to the three types of sheet described previously.
(2) Furnace temperature (for pre-heating sheet).
(3) Variant of 'Vydax'.
(4) A naphthenic spindle oil + 3% oleic acid.

APPENDIX I

PHASE IV WORK SCHEDULE

Phase IV extends over a period of five months and consists of production and testing of 12" wide foil in the three columbium base alloys, the two tantalum base alloys and in pure tungsten.

The 0.100" thick columbium alloy and tantalum alloy sheets will be rolled under tension to approximately 0.020" thickness and annealed in a continuous vacuum heat treatment furnace. Some of the annealed strip will be rolled directly to 0.005", one of the target thicknesses for the contract. The remainder of the 0.020" thick material will be rolled to 0.003", 0.002" and 0.001" thicknesses with an intermediate anneal interposed in the thickness range 0.004" - 0.007". Portions of the finished strip will be heat treated.

Tungsten sheet, 0.020" - 0.060" thick x 12" wide will be purchased for rolling at D.M.C. and rolled to 0.005" and below with intermediate stress relief anneals. The minimum gage tungsten strip attainable on D.M.C. equipment cannot be accurately predicted at this time.

APPENDIX II

GAGE SURVEYS OF 0.100" SHEETS

Gage uniformity and flatness data are given for each sheet. Ten thickness measurements are given for each sheet; five measurements across the width of the sheet at two locations. The two outside measurements in all cases are on the sheet edges and the other readings at equally spaced intervals across the width. The distances of the transverse thickness profiles from the ends are to be taken as distances from the ends of the 'fish-tails'. Thickness profiles in the centers of the sheets are more uniform than at the locations of measurements recorded here.

12"	12"
0.0905"	0.0878"
0.0930"	0.0912"
0.0928"	0.0915"
0.0930"	0.0918"
0.0934"	0.0924"

Flatness: Length 1/16", Width 1/32"

Sheet D-43-1

12"	12"
0.0938"	0.0942"
0.0970"	0.0958"
0.0954"	0.0972"
0.0954"	0.0963"
0.0941"	0.0949"

Flatness: Length 1/8", Width 1/8"

Sheet D-43-2

12"	12"
0.1018"	0.1011"
0.1039"	0.1026"
0.1039"	0.1019"
0.1038"	0.1016"
0.1005"	0.0986"

Flatness: Length Width

Sheet D-43-3

6"	6"
0.0952"	0.0921"
0.0979"	0.0964"
0.0979"	0.0962"
0.0978"	0.0959"
0.0961"	0.0935"

Flatness: Length 5/16", Width 1/8"

<u>Sheet B-66-1</u>

6"	6"
0.0925"	0.0939"
0.0961"	0.0960"
0.0959"	0.0960"
0.0959"	0.0909"
0.0938"	0.0938"

Flatness: Length Width

<u>Sheet B-66-2</u>

12"	12"
0.0945"	0.0944"
0.0962"	0.0959"
0.0970"	0.0965"
0.0965"	0.0959"
0.0942"	0.0936"

Flatness: Length 7/32", Width 1/8"

<u>Sheet B-66-3</u>

← 12" →	← 12" →
0.0892"	0.0880"
0.0916"	0.0899"
0.0922"	0.0890"
0.0918"	0.0890"
0.0898"	0.0870"

Flatness: Length 5/32", Width 1/16"

<u>Sheet Cb-752-1</u>

← 12" →	← 12" →
0.0859"	0.0870"
0.0891"	0.0899"
0.0889"	0.0900"
0.0884"	0.0899"
0.0873"	0.0892"

Flatness: Length 1/2", Width 1/8"

<u>Sheet Cb-752-2</u>

← 12" →	← 12" →
0.0969"	0.0932"
0.0978"	0.0958"
0.0998"	0.0968"
0.0985"	0.0970"
0.0962"	0.0968"

Flatness: Length 5/16", Width 3/32"

<u>Sheet Ta-10%W-1</u>

← 12" →	← 12" →
0.0950"	0.0978"
0.0985"	0.0980"
0.0985"	0.1000"
0.0980"	0.0998"
0.0952"	0.0958"

Flatness: Length 1/16", Width 1/32"

<u>Sheet Ta-10%W-2</u>

← 12" →	← 12" →
0.1019"	0.1022"
0.1035"	0.1038"
0.1038"	0.1035"
0.1039"	0.1038"
0.1000"	0.1010"

Flatness: Length 1/32", Width 1/32"

<u>Sheet Ta-10%W-3</u>

← 12" →	← 12" →
0.1005"	0.1017"
0.1017"	0.1032"
0.1021"	0.1031"
0.1025"	0.1038"
0.1004"	0.1006"

Flatness: Length 3/8", Width 3/32"

<u>Sheet T-111-1</u>

12"	12"
0.0992"	0.0990"
0.1018"	0.1021"
0.1017"	0.1018"
0.1014"	0.1010"
0.0998"	0.1001"

Flatness: Length 1/4", Width 1/8"

<u>**Sheet T-111-1**</u>

APPENDIX III

RECENT DATA ON C-129 ALLOY

The Phase I Report under this contract (State-of-the Art Survey) gave detailed information on seven columbium base alloys including the Wah Chang C-129 alloy (Cb-10%W-10%Hf). Since publication of that report further information on C-129 alloy became available in a paper by R. T. Torgerson, The Boeing Company ('Development and Properties of Columbium-10% Tungsten-10% Hafnium Alloy') at the 1962 AIME Fall Meeting in New York City.

In our State-of-the-Art Survey it was implied that weld ductility in C-129 alloy could only be obtained by post-weld heat treatment. Data published in the above paper show that fusion welds in good quality C-129 alloy sheet are ductile in the as-welded condition using either the TIG or EB welding process. Bend ductility figures for the C-129 alloy are given in the following three tables which are extracted from the Boeing paper.

BEND DUCTILITY OF TIG WELDED C-129 ALLOY

Specimen No.	Specimen Type	Base Metal Thickness Inches	Weld Thickness Inches	Ram Radius Inches	Ratio of Ram Radius to Thickness	Face or Root Bend	Results
B-1	Type E	0.0206	0.0236	0.142	6T	Face	Brittle fracture
B-2	Type E	0.0206	0.0243	0.142	6T	Face	Brittle fracture
B-3	Type E	0.0206	0.0242	0.242	10T	Face	Ductile >90^{o}
C-1	Type F	0.0206	0.0231	0.142	6T	Face	Ductile >90^{o}
C-2	Type F	0.0206	0.0237	0.142	6T	Root	Ductile >90^{o}
C-3	Type F	0.0206	0.0229	0.047	2T	Face	Cracked
C-4	Type F	0.0206	0.0227	0.090	4T	Face	Cracked

a) Ram rate 10 in/min., bend angle 90^{o} to 105^{o} after springback.

b) Specimens prepared by butt welding with no surface finishing of weld bead.

c) Base metal: C-129 Alloy Heat C
0.020-inch sheet
Fully recrystallized condition (1 hour at $2400^{o}F$)

BEND DUCTILITY OF ELECTRON BEAM WELDED C-129 ALLOY

Specimen No.	Specimen Type	Welding Passes	Base Metal Thickness	Ram Radius Inches	Ratio of Ram Radius to Thickness	Face or Root Bend	Results
4-1	Type E	Single	0.0206	0.20	10T	Face	Ductile >90°
4-2	Type E	Single	0.0206	0.12	6T	Face	Ductile >90°
4-3	Type E	Single	0.0206	0.08	4T	Face	Ductile >90°
4-4	Type E	Single	0.0206	0.02	1T	Face	Ductile >90°
4-5	Type E	Single	0.0206	0.02	1T	Face	Cracked
6-1	Type E	Double	0.0206	0.20	10T	Face	Ductile >90°
6-2	Type E	Double	0.0206	0.12	6T	Face	Ductile >90°
6-3	Type E	Double	0.0206	0.08	4T	Face	Ductile >90°
6-4	Type E	Double	0.0206	0.08	4T	Face	Ductile >90°
6-5	Type E	Double	0.0206	0.02	1T	Face	Ductile >90°
6-6	Type E	Double	0.0206	0.02	1T	Root	Cracked
2-1	Type E	Double	0.0206	0.03	1.5T	Face	Ductile >90°
2-2	Type E	Double	0.0206	0.03	1.5T	Face	Ductile >90°
2-3	Type E	Double	0.0206	0.03	1.5T	Face	Ductile >90°
2-4	Type E	Double	0.0206	0.03	1.5T	Root	Ductile >90°
2-5	Type E	Double	0.0206	0.03	1.5T	Root	Ductile >90°
2-6	Type E	Double	0.0206	0.03	1.5T	Root	Ductile >90°

a) Ram rate 10 in/min., bend angle 90° to 105° after springback.

b) Specimens prepared by butt welding with no surface finishing of weld bead.

c) Base metal: C-129 Alloy Heat C
0.020-inch sheet
Fully recrystallized condition (1 hour at 2400°F)

EFFECT OF HEAT TREATMENT ON BEND DUCTILITY OF TIG WELDED C-129 ALLOY

Post-Weld Heat Treatment	Bend Angle	Ram Rate	Minimum Bend Radius	Remarks
As-Welded	105°	10"/min.	10T	Fractured at 8T
8 hrs. @ 1850°F	105°	10"/min.	4T	Cracked at 3T
1 hr. @ 2200°F	105°	10"/min.	4T	Cracked at 2T
1 hr. @ 2200°F	105°	10"/min.	2T	Not tested at lower radius
1 hr. @ 2400°F	105°	10"/min.	1T	Not tested at lower radius

a) Specimen Type E (weld parallel to bend axis).

b) Specimens prepared by butt welding and grinding weld bead flush with base metal.

c) Heat treatment conducted in a vacuum of 1×10^{-4} mm Hg or lower.

d) Specimens tested at room temperature.

e) Base metal: C-129 Alloy Heat B
0.030-inch sheet
Fully recrystallized condition
(1 hour at 2400°F)

E.I. duPont de Nemours & Co., Inc.
Metal Products
Pigments Department
Wilmington, Delaware

REFRACTORY ALLOY FOIL ROLLING DEVELOPMENT PROGRAM

May, 1963

ASD Project No. 7-987
(Contract AF33(657)-8912)
Phase III, Unclassified Report

The processing of five alloys from ingot to 12" wide x 0.100" sheet is described. The five
(over)

UNCLASSIFIED
1. Extrusion
2. Hot-rolling
3. Conditioning
4. Cold-rolling
5. Tungsten rolling

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